Solid phase extraction using non-polar and polar Magic Chemisorber®

4. Headspace analysis of silicone rubber

[Background] Headspace solid phase extraction (HS-SPE) using non-polar Magic Chemisorber ® MC-S500 and polar Magic Chemisorber ® MC-PEG-S is described for the analysis of volatile components in silicone rubber.

[Experimental] A non-polar Magic Chemisorber® MC-S500 (film thickness of PDMS: 500 µm) was attached to an Eco-Stick DF and held in the headspace of a 17 mL vial containing 50 mg of silicone rubber (used stretchable food cover) at 80 ºC for 10 min. The Magic Chemisorber® was then set in the pyrolyzer furnace and heated: 100 - 230 ºC (3 min hold). Thermally desorbed compounds were swept by the helium carrier gas to a GC separation column (UA-WAX) and cryo-trapped at the head of the separation column using a MicroJet Cryo-Trap. Then, the trap was turned off, and the released volatiles were separated and detected by a quadrupole mass detector. For comparison, the analysis was similarly performed using the polar Magic Chemisorber® MC-PEG.

[Results] Chromatograms of the extracted compounds from the silicone rubber are shown in Fig. 1, and peak assignments are summarized in Table 1. Non-polar Magic Chemisorber® mainly extracted cyclic siloxanes (1-7). On the other hand, with the polar Magic Chemisorber®, almost no cyclic siloxanes (2-6) were extracted. As demonstrated above, volatile components in silicone rubber can be easily and quickly analyzed using solid-phase extraction with Magic Chemisorber ®, followed by thermal desorption GC/MS with a pyrolyzer.

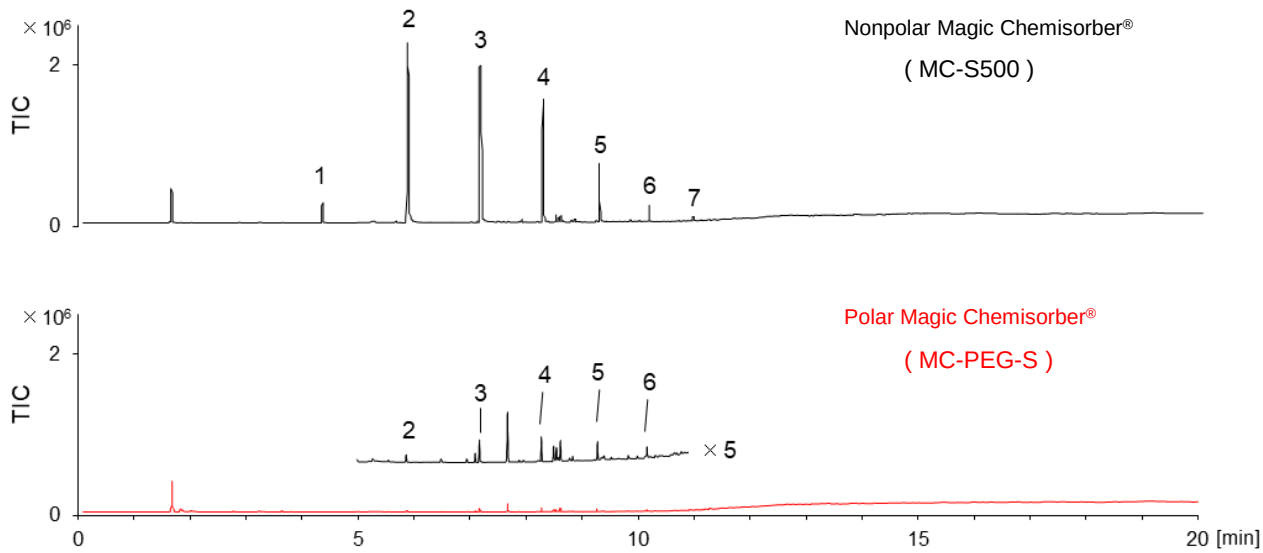

Fig. 1 Chromatograms of components extracted from silicone rubber by polar and nonpolar Magic Chemisorber®

Sample amount: 50 mg, Headspace extraction: 10 min at 80 ºC, Thermal desorption temp.: 100 - 230 ºC (40 ºC/min, 3 min hold),
Cryo-trapped with MicroJet Cryo-Trap, Separation column: Ultra ALLOY-WAX (polyethylene glycol), *L* = 30 m, i.d. = 0.25 mm, df = 0.25 µm
Column flow rate: 1 mL/min, Split ratio: 1/10, GC oven temp.: 40 ºC (2 min hold) - 230 ºC (20 ºC/min, 8.5 min hold)

Table 1 Compounds extracted from Silicone rubber

#	Compound	#	Compound	#	Compound
1	Octamethyl-cyclotetrasiloxane(D4)	4	Tetradecamethyl-cycloheptasiloxane(D7)	7	Eicosamethyl-cyclodecasiloxane(D10)
2	Decamethyl-cyclopentasiloxane(D5)	5	Hexadecamethyl-cyclooctasiloxane(D8)		
3	Dodecamethyl-cyclohexasiloxane(D6)	6	Octadecamethyl-cyclononasiloxane(D9)		

Keywords : Evolved gas analysis, Solid-phase extraction device, Thermal desorption GC/MS, Silicone rubber

Products used : Multi-functional pyrolyzer, Magic Chemisorber® MC-S500, Magic Chemisorber® MC-PEG, MicroJet Cryo-Trap, UA-WAX

Applications : Food container, Volatiles analysis

Related technical notes : MCA-020E, MCA-021E

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